

NPS ARCHIVE
1969
WHITE, D.

THE EFFECT OF THE INTERFACE COMPOUND
ON THE TENSILE STRENGTH OF AN
ALUMINUM MATRIX-BORON FIBER COMPOSITE

DAVID A. WHITE

MAY 1969

COURSE 13A.

Thesis
W5508

12-0001
FEDERAL POST OFFICE
MONTEREY, CALIF. 93940

THE EFFECT OF THE INTERFACE COMPOUND ON THE
TENSILE STRENGTH OF AN ALUMINUM MATRIX-
BORON FIBER COMPOSITE

by

DAVID A. WHITE

S. B. U. S. COAST GUARD ACADEMY (1958)

Submitted in Partial Fulfillment of the Requirements for the
Master of Science Degree
in Mechanical Engineering
and the Professional Degree, Naval Engineer
at the
Massachusetts Institute of Technology

THE EFFECT OF THE INTERFACE COMPOUND ON THE
TENSILE STRENGTH OF AN ALUMINUM MATRIX-
BORON FIBER COMPOSITE

by

DAVID A. WHITE

Submitted to the Department of Naval Architecture and Marine Engineering on 23 May 1969 in partial fulfillment of the requirements for the Master of Science Degree in Mechanical Engineering and the Professional Degree, Naval Engineer.

ABSTRACT

This study was conducted to determine the effect of interface compound on the strength of aluminum-boron composites. Cylindrical composites were prepared by vacuum infiltration at various liquid aluminum temperatures to promote the formation of varying amounts of interface compound. The tensile strength of these composites was found to follow a modified law-of-mixtures rule which takes into account only the actual volume fractions of fiber and matrix and neglects the volume fraction of the compound, however, test strengths were only $2/3$ of modified predicted strengths. This strength variation is attributed to composite preparation imperfections. Compound formation did not prevent stress transfer from fiber to fiber through the matrix.

Thesis Supervisor: J. W. MAR

Title: Professor of Aeronautics and Astronautics

TABLE OF CONTENTS

<u>Section Number</u>		<u>Page Number</u>
	ABSTRACT	ii
	LIST OF ILLUSTRATIONS AND TABLES	iv
	ACKNOWLEDGEMENTS	v
	INTRODUCTION.	1
I	PROCEDURES	6
II	RESULTS.	13
III	DISCUSSION OF RESULTS	31
IV	CONCLUSIONS.	37
V	RECOMMENDATIONS	38
	BIBLIOGRAPHY	39

LIST OF ILLUSTRATIONS AND TABLES

<u>Figure Number</u>		<u>Page Number</u>
1	Oven Treated Specimen	7
2	Apparatus For Vacuum Infiltration	10
3	Instron Recorder Trace	17
4	Stress-Strain Curves	18
5	Stress-Strain Curves	19
6	Complete Cross Section, 35X	20
7	Complete Cross Section, 35X	21
8	Complete Cross Section, 40X	22
9	Cross Sections of Single Fiber in Matrix, 500X	24
10	Cross Sections of Single Fiber in Matrix, 500X	25
11	Cross Sections of Fibers in Matrix, 500X	26
12	Longitudinal View of Fibers in Matrix, 50X	27
13	Longitudinal View of Fibers in Matrix, 100X	29
14	Longitudinal View of Fiber in Matrix, 300X Compound Formation Can Be Seen on the Fiber	30
15	Curve of Normalized Strength to Diameter Ratio Squared. .	34
 <u>Table Number</u>		
I	Specimen Preparation Summary	14
II	Tensile Test and Metallography Summary	15

ACKNOWLEDGEMENTS

The author wishes to express gratitude to Professor James W. Mar and Dr. Lawrence A. Shepard for the suggestion of this thesis topic and for their advice and encouragement. Especially appreciated was the day to day counsel and patient assistance provided by Dr. Shepard.

Mr. Gregory Garmong is thanked for providing valuable technical aid and the assistance and advice in laboratory techniques of Mr. Guenter Arndt and Mr. I. Puffer were greatly appreciated.

Finally, the clerical assistance and loving help provided by my Wife, Janet, in preparing this thesis is acknowledged.

INTRODUCTION

A composite material can be broadly defined as a combination of at least two distinct materials which retain their identities in the combined form and yield a material which exhibits properties not possessed by the constituents acting alone. A common example is fiberglass-reinforced plastics such as those widely used in the boat building industry. Discounting the ancients use of mud-straw building bricks, laminated bows, and the like, development of composite materials is a recent phenomenon. Serious efforts toward developing a modern technology of composite materials began less than two decades ago.

The majority of modern composite materials developed thus far have been aimed at increasing mechanical properties such as strength-to-weight ratio, stiffness, toughness and high-temperature strength. Broutman and Krock¹ have classed composites of this nature into three basic types: dispersion-strengthened, particle-reinforced and fiber-reinforced. Many fiber-reinforced composites created to date have exhibited very high uniaxial strength characteristics in an industrially useable or potentially useable material. This is achieved by using extremely strong and often, brittle fibers not ordinarily thought of as industrial materials embedded in a weaker more ductile matrix material which is more readily adaptable to industrial use. The high strength is achieved by transfer of stress through the matrix to all of the fibers loading them uniformly to fracture.

The law-of-mixtures relation applies to fiber composites for tensile strengths if optimum preparation conditions are attained.

Continuous or sufficiently long discontinuous aligned fibers, parallel to the tensile axis and firmly bonded to the matrix so that no slippage can occur and stress can be transferred at the interface are conditions which can produce law-of-mixtures strengths. When the foregoing conditions are met, the composite, fiber and matrix strains are equal and the maximum composite stress can be represented by:²

$$\sigma_c = \sigma_f V_f + \sigma_m' V_m \quad (1)$$

where

σ_c	ultimate tensile stress of composite
σ_f	ultimate tensile stress of fibers
σ_m'	matrix stress when fibers are strained to their ultimate tensile strain
V_f	volume fraction of fibers
V_m	volume fraction of matrix; $V_f + V_m = 1$

McDanels, Jech, and Weeton³ and a number of subsequent workers have experimentally verified the law-of-mixtures in metal matrix composites. The McDanels' group prepared copper-tungsten composites, containing different volume fractions of fibers, by infiltration of copper into bundles of tungsten fibers. These composites were then tensile tested at room temperature and compared against calculated law-of-mixture strengths with good agreement.

For equation (1) to apply to a fiber composite it is necessary that a minimum fiber volume fraction be exceeded. This minimum is defined by:

$$V_{f \text{ MIN.}} = \frac{\sigma_m - \sigma_m'}{\sigma_f + \sigma_m - \sigma_m'} \quad (2)$$

where:

σ_M ultimate tensile stress of matrix

Below this minimum fiber volume fraction there is no real fiber strengthening. Failure of all the fibers does not produce composite failure, and the net composite strength becomes $\sigma_M V_M$. Furthermore, if the fiber composite is to be stronger than the matrix material alone, a critical fiber volume fraction must be exceeded. This is defined as:

$$V_{f \text{ CRIT.}} = \frac{\sigma_M - \sigma'_M}{\sigma_f - \sigma'_M} \quad (3)$$

As can be seen by comparison of (2) and (3) $V_{f \text{ min.}}$ is always less than $V_{f \text{ crit.}}$ The foregoing considerations are based upon the assumptions that the fibers do not alter the stress-strain characteristics of the matrix material, the fibers are of equal strength and all break in a given cross section.¹

Another important consideration in fiber composites is the mutual reactivity by the constituents. Using tungsten fibers and copper binary alloys containing elements having varying solubility in tungsten as the matrix material, Petrusek⁴ found that the ultimate tensile strength of the unalloyed fiber composites was generally higher than that of alloyed fiber composites. This reduction in strength increased with increasing penetration of the alloying element into the tungsten fiber. Heitman⁵ found that room temperature strength of reacted molybdenum-aluminum composites could be predicted by a modified mixture rule which neglects the strength contribution of the brittle compound.

The aluminum-boron composite system is particularly attractive as a light weight structural material because it combines high strength

to density and high modulus to density ratios. Boron wires of 4-mil diameter have a density of 0.094 lb/in^3 and can be manufactured today with an average tensile strength of 400,000 psi and an average modulus of 60,000,000 psi⁶. Aluminum has a density of 0.0975 lb/in^3 ⁷. Boron wires are manufactured by a chemical vapor-plating process with the boron being deposited on a 0.0005-in. tungsten wire substrate⁶.

Estes⁸ studied aluminum-boron composite preparation techniques by various drawing processes. His work indicated that dip brazing of aluminum alloy tubes containing boron wires followed by hot drawing resulted in 100% dense specimens with little fiber breakage during preparation. However, tensile tests on these specimens showed strengths less than those predicted by the law-of-mixtures. These lower strengths were attributed to imperfect bonding of the constituents.

Boron is known to react with aluminum at high temperatures resulting in degradation of the boron filaments⁹. Camahort reported aluminum attack on boron filaments using single filaments immersed in aluminum powder at 600°C, 700°C, and 800°C⁹. Alexander¹⁰ prepared aluminum-boron composites by liquid infiltration. He found that boron filaments exposed to liquid aluminum for ten minutes at 700°C exhibited a scalloped circumference but no observable reaction layer. He also reported that no dissolution of the wire nor formation of reaction layer was observed in aluminum-boron composites held at 500°C and 600°C for one hour.

Wolff and Hill¹¹ did extensive work on boron filament-metal matrix composites. Among their conclusions for aluminum-boron

composites of continuous fibers prepared by vacuum infiltration was that aluminum temperatures between 720°C and 800°C resulted in the highest values of tensile strength and the absence of voids. Below this temperature range the fibers failed to be properly wet and above this range circumferential cracks developed in the fibers as well as gross dissolution of the boron, regardless of heating time. They also reported that boron fibers in aluminum matrices held at 500°C for up to 112 hours showed no degradation that is perceptible at a magnification of 250X.

Aluminum-boron composites have been tested for use in inlet structures of ramjet engines and for applications in turbine compressors¹². Pratt & Whitney Aircraft has recently operated a JT8D engine containing an experimental first stage fan equipped with aluminum-boron blades¹³. The Office of Naval Research of the Navy Department has expressed an interest in composite materials for the potential weight saving realizable in many naval applications, particularly as submersible pressure hulls¹⁴. One of the problem areas involved in applications of aluminum-boron composites is the strength degradation resulting from reaction at the interface. This study will be directed toward developing an understanding of the strength degradation.

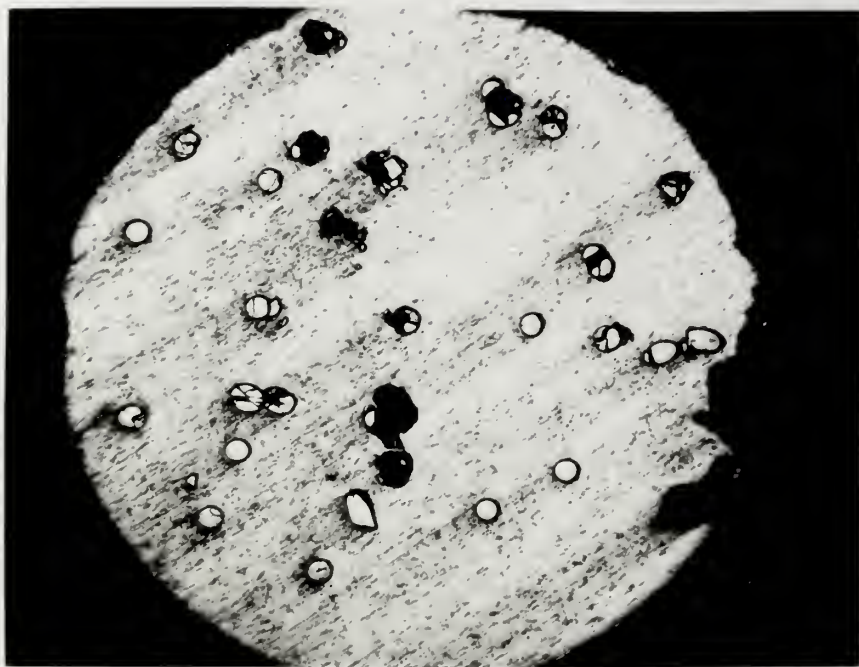
I. PROCEDURES

Fiber composites were prepared using high purity (99.99+%) aluminum as matrix material and 0.00395 inches diameter boron filaments manufactured by United Aircraft. Cylindrical tensile test specimens were made by vacuum infiltration. This method was selected because of recent reports of success^{11, 12} and the fact that it can be easily carried out in the laboratory with a minimum of equipment and apparatus. It was originally planned to heat treat these specimens in the solid state in an oven for various times at given temperatures to produce varying amounts of interface compound prior to tensile testing. One specimen was encapsulated in an evacuated vycor tube and placed in a 600°C oven for 64 hours. Microscopic examination of a polished cross section of this specimen did not reveal identifiable amounts of interface compound. The specimen was then re-encapsulated and heat treated at 650°C for 24 hours. A malfunction of the oven temperature control caused the temperature to approach 660°C during the next 16 hours. The temperature was then reduced to 580°C for 53 hours. In summary, the temperature history for this specimen is 580°C or above for 157 hours and 650°C or above for 40 hours. Microscopic examination of a polished cross section revealed only minor attack, evidenced by minor circumferential scalloping of the boron filaments. Figure 1 shows a polished cross section of this specimen after 157 hours in the oven.

Since aluminum in the solid form demonstrated relative non-reactivity with the boron filaments, even at temperatures close to the melting point, it was decided to attempt to produce interface



A. INDIVIDUAL FIBER, 500X



B. COMPLETE CROSS SECTION, 45X

FIGURE 1. OVEN TREATED SPECIMEN

compound during the infiltration process. This was done by using various temperatures and times for the boron fibers to be in contact with liquid aluminum. Tensile testing and microscopic examination followed the infiltration process.

A. Specimen Preparation.

Composite specimens were prepared using an electric resistance furnace, vacuum pump, 2mm nominal inside diameter quartz tube, and a graphite crucible. The apparatus arrangement is shown in Figure 2. Pieces were cut to crucible size from a high purity aluminum ingot, soaked in trichloroethylene, and then surface oxides were ground off.

The fibers were hand cut to 3-inch nominal lengths from a roll of boron filament and cleaned with 4/0 emery paper. These wires were soaked in trichloroethylene and then individually dipped into molten aluminum at 720°C so that they were "tinned" throughout their entire length with aluminum. "Tinning" was not employed in initial attempts to make these specimens with the result that the boron fibers were displaced to the sides during infiltration and exposed on the surface of the specimen. Wolff and Hill¹¹ found similar orientation behavior in low density specimens prepared in a similar manner.

The quartz tubes were cut to eight inch lengths and carbon coated using colloidal graphite in water and then dried over a bunsen burner or placed in a 110°C oven to dry.

The "tinned" fibers were given a final bath in methanol and 30 fibers were individually placed in a carbon coated quartz tube. A length of chromel wire with a small piece of aluminum foil over its end was inserted into the other end of the quartz tube to prevent the

fibers from traveling up the tube during vacuum pumping and when the molten aluminum entered the tube, and to promote freezing of the aluminum. Vacuum was drawn on the tube with the end containing the fibers being sealed off with a piece of aluminum foil. The charge and crucible were preheated in the furnace prior to melting to cause drying. The tube, under vacuum, was then lowered to a depth which covered the fibers into the molten aluminum, which was at various temperatures from 720°C to 800°C, and held there for given times. Immediately upon entering the molten aluminum, the foil seal, as shown in Figure 2, on the end of the tube melted and aluminum was drawn into the tube freezing at the base of the chromel wire. This point was held just above the level of the molten aluminum.

Upon completion of time in the crucible the quartz tube was slowly withdrawn and allowed to cool in the air at room temperature. Removal of the completed specimen from the quartz tube presented some difficulties since in spite of the carbon coating there was some reaction between the aluminum and quartz at various points inside the tube. This presumably occurred in spots where carbon had not adhered to the inside of the tube. Surface imperfections resulted where reactions occurred. Use of graphite tubes in lieu of quartz would probably eliminate this difficulty.

B. Tensile Testing.

Composite specimens removed from the quartz tubes were prepared for tensile testing by cutting off the ends of the specimens where obvious imperfections were observed. In general, specimens were non-cylindrical at the lower end, probably due to molten aluminum

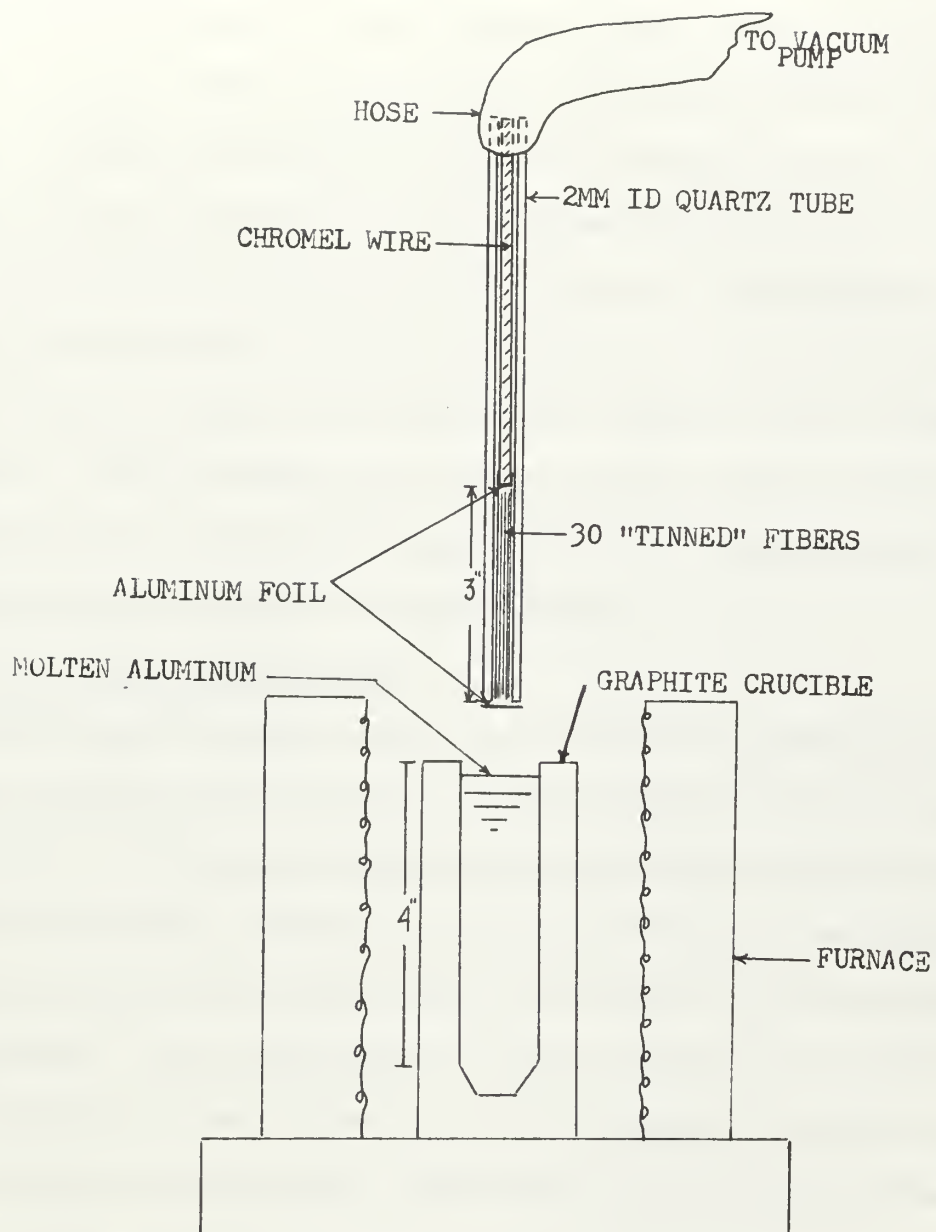


FIGURE 2. APPARATUS FOR VACUUM INFILTRATION.

running out of the tube during removal from the crucible. Upper end mal-formations existed on some specimens and are believed to be the result of rapid surface freezing prior to full infiltration of this area. It should be recalled that this area was held just above the level of molten aluminum and is also influenced by the wire inserted from the top of the tube acting as a heat sink. After being cut to length, aluminum grips 0.57 inches in diameter and either 1/4, 1/2 or 3/4 inches in length were bonded to the specimens using an epoxy adhesive. Tensile testing was done on an Instron machine at room temperature with the crosshead traveling at 0.02 inches per minute. Crosshead travel was used to determine strain.

C. Polishing and Metallography.

After tensile testing a piece was cut from each specimen using either a carbide cutting wheel or a diamond cutting wheel and mounted in bakelite. Both cutting wheels caused considerable damage to the surface to be viewed. Fibers were chipped and split by the cutting wheels and pieces were transferred to and imbedded in various places in the matrix. The cutting wheels are also considered to be responsible for creating void areas around the wires by whipping or pushing the wires through the soft matrix. These void areas could be polished out as described below.

Various polishing schemes were tried in an attempt to achieve a flat surface. First, silicon carbide paper and emery paper were used progressing from coarse to fine. This procedure was considered unsatisfactory since fiber breakage occurred and void areas in the matrix were once again created.

Diamond paste on a rotating wheel was tried next using the following micron sizes in order: 45, 15, 6, 3, and 1. This procedure did not break fibers or create voids but the 45 and 15 micron pastes did not polish out the voids created by the cutting wheels. The 6 micron paste did polish out the voids but was a slow process due to the fine size.

It was found that by using 600-A silicon carbide paper initially and sparingly, then polishing on 6 micron diamond paste a satisfactory surface could be produced in reasonable time. Further polishing on finer diamond paste generally produced a clean matrix surface, but polished the matrix below the fiber surface and rounded the fiber edges.

Microscopic examinations of the polished specimens were done on a Reichert optical metallograph to determine fiber distributions and wire diameters in the composites. Photographs of cross sections of each specimen were made. Fiber diameters in each specimen were determined by photographs taken at 500X. Actual magnification of each specimen was determined by comparison with a gage scribed at 0.01mm intervals.

II. RESULTS

Table I summarizes the preparation conditions and final configuration of the ten tensile specimens. The infiltration temperature range used to prepare specimens and to promote interface compound formation agreed with that found to be optimum by Wolff and Hill¹¹, namely, 720°C to 800°C. Specimens prepared at 800°C exhibited surface pitting. This is believed to be the result of aluminum-quartz reaction during the infiltration process. The gage length used in the tensile test of these specimens was cut from the best section of the 3-inch length prepared by infiltration. The 720°C specimens exposed to liquid aluminum for short times exhibited extremely poor surface conditions. Specimens I and J were so bad that a useable sound section could not be cut from the infiltration prepared 3-inch lengths. The specimens were tensile tested with the surface imperfections present. Other variations in gage lengths resulted from the different grip lengths used and varying amounts of end mal-formation which were cut away. Specimen diameters shown are those measured on the actual gage length. Variations are due to non-uniformity of the quartz tubes used.

The specimens in this table are listed in decreasing order of expected severity of aluminum attack on the boron fibers. Previous work^{9, 11} indicated that attack generally increased with time and temperature.

Table II summarizes the results of tensile testing and metallographic examination. Specimens B and D were tensile tested twice. After the first test a new grip was attached to the largest

TABLE I
SPECIMEN PREPARATION SUMMARY

SPECIMEN	MELT TEMP. DEG C.	TIME IN CRUCIBLE	DIAMETER INCHES	GAGE LENGTH INCHES	REMARKS
A	800	6 min.	0.087	0.875	Poor surface areas cut off.
G	800	3 min.	0.087	0.975	Poor surface areas cut off.
B	750	1 min.	0.088	1.437	
C	740	1 min.	0.089	1.563	
E	720	2 min.	0.086	1.312	
F	720	2 min.	0.087	1.124	
D	720	30 sec.	0.087	1.360	
H	720	10 sec.	0.088	0.873	Poor surface areas cut off.
I	720	10 sec.	0.084	1.818	Poor surface formation throughout with fibers visible.
J	720	5 sec.	0.089	1.188	Surface voids near mid- length with fibers visible at voids.

TABLE II
TENSILE TEST AND METALLOGRAPHY SUMMARY

SPECIMEN	MAX. STRESS PSI	NOMINAL STRENGTH PSI	MEASURED FIBER DIAMETER 10-3 INCHES	MATRIX VOIDS IN FRACTURE SURFACE	FIBER ORIENTATION
A	9,520	20,260	3.39	Center	Throughout
G	8,900	20,260	3.61	None	Grouped
B ₁	9,600	19,840	3.78	Center	Throughout
B ₂	9,845	19,840	3.78	Center	Throughout
C	11,820	19,450	3.78	None	Well Distributed
E	12,980	20,650	3.82	None	Well Distributed
F	10,920	20,260	3.78	Periphery	Peripheral
D ₁	8,280	20,260	3.79	Center	Peripheral
D ₂	10,360	20,260	3.79	None	Peripheral
H	13,020	19,840	3.88	Periphery	Throughout
I	9,210	21,600	3.86	Periphery	Throughout, Poor bond
J	13,150	19,450	3.85	Periphery	Grouped

portion of the broken specimen. The "nominal strengths" shown in the table for each specimen are based upon the law-of-mixtures and original boron filament diameter. A nominal strength for the boron filaments of 300,000 psi was used in this calculation and stress-strain curves of Trozera, Sherby and Dorn¹⁵ for annealed high purity aluminum were used to determine the aluminum matrix stress.

Figure 3 is an actual trace from the Instron recorder of the test on specimen H. The ripples appearing in the trace after maximum load was attained represent fiber breaks which were audible to an observer standing within several inches of the specimen. All specimens exhibited this characteristic ripple trace after achieving maximum load. Both specimens A and E, exhibited fiber breaks prior to achieving maximum load. There were two audible breaks which were seen on the trace prior to maximum loading on specimen E and one on specimen A. During the tensile test of specimen I, the fibers pulled completely out of the matrix at the fracture surface indicating extremely poor bonding.

Calculated stress-strain curves for all tensile tests are shown in Figures 4 and 5.

Photographs of the complete cross section of each specimen showing the fiber orientation resulting from infiltration are contained in Figures 6 through 8. Fiber distributions vary in character from specimen to specimen. Some are generally well distributed throughout the matrix such as in specimens C and E. Others show a tendency for the fibers to seek a circumferential or peripheral orientation in the

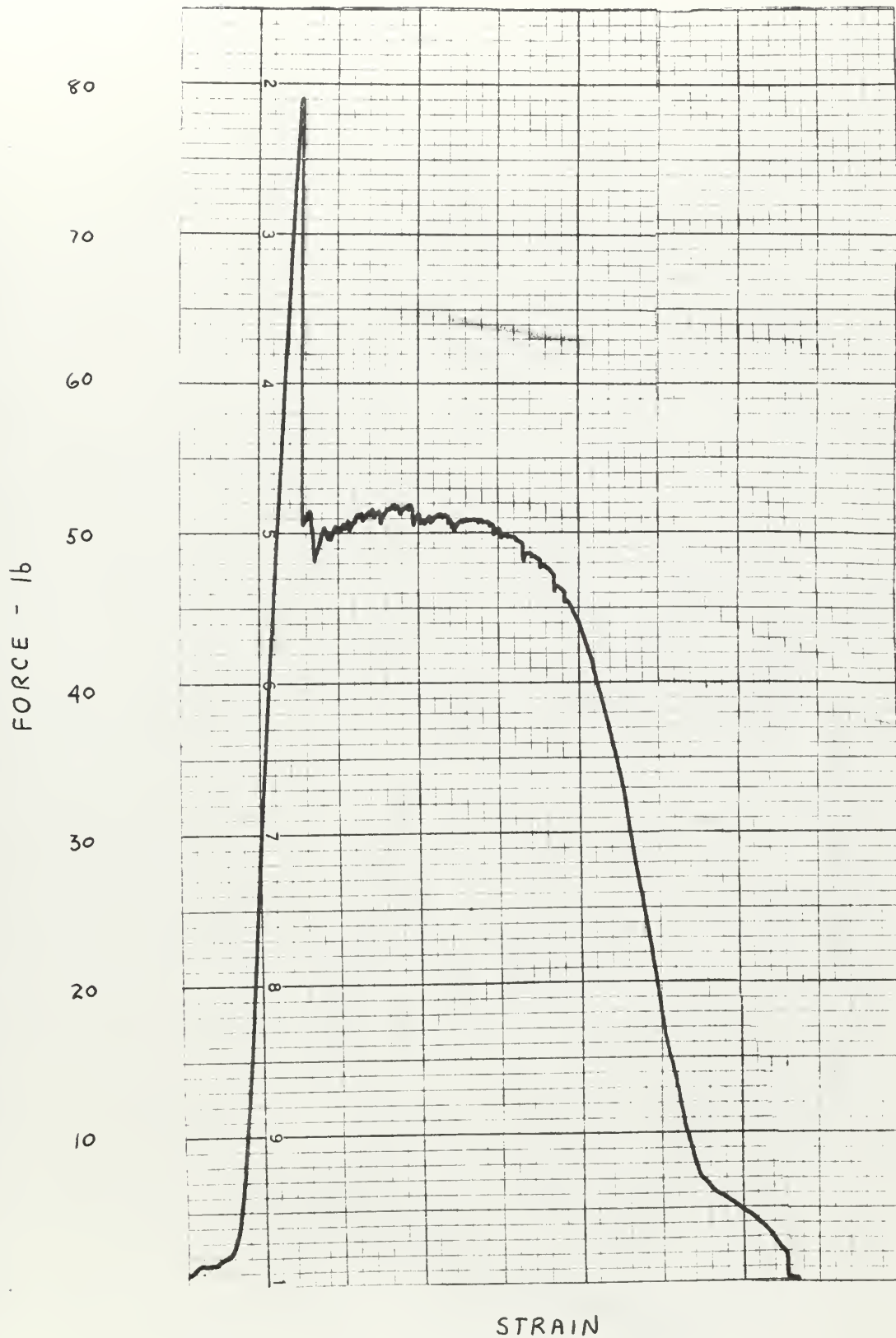


FIGURE 3 INSTRON RECORDER TRACE



FIGURE 4. STRESS-STRAIN CURVES

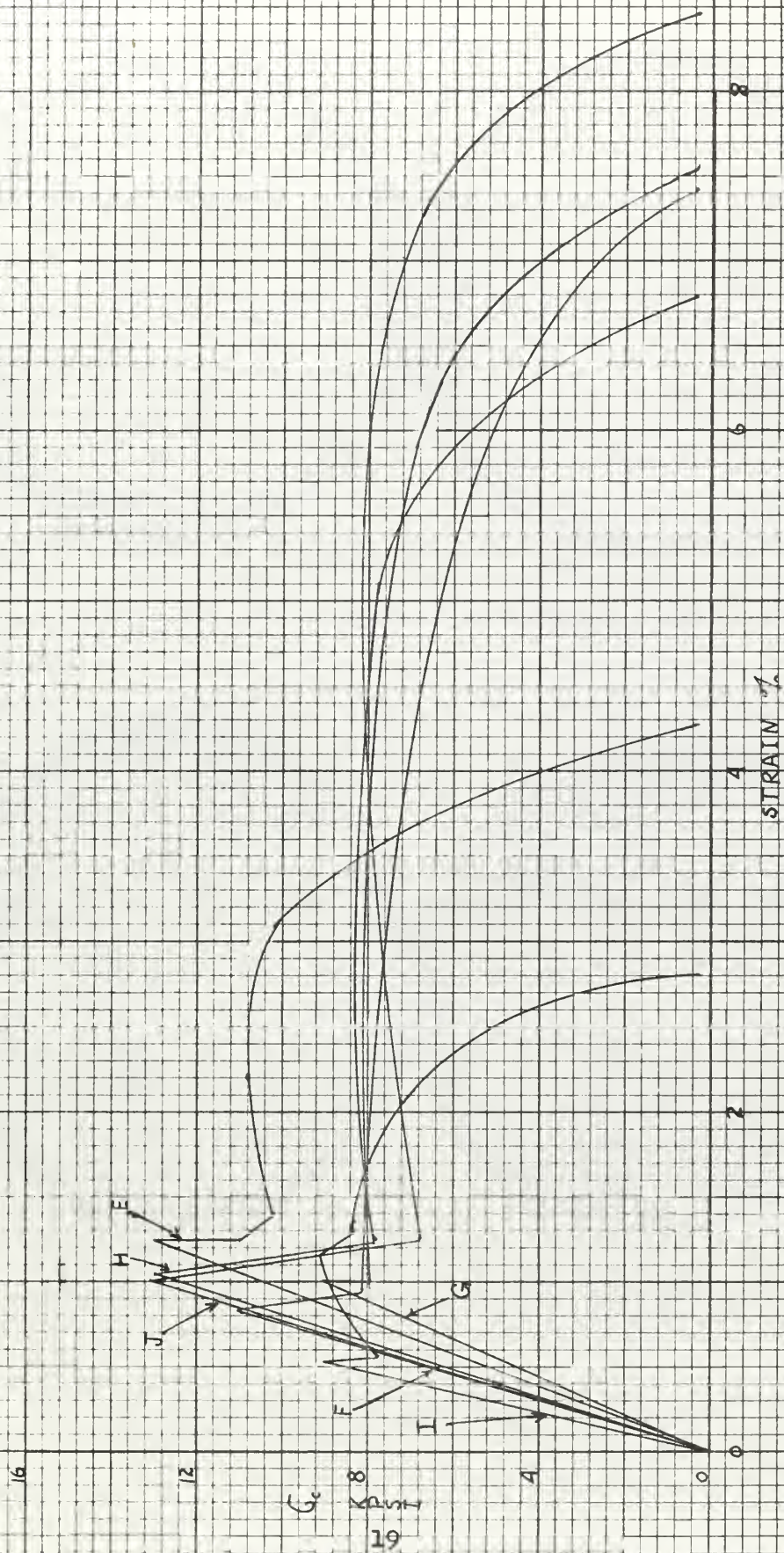
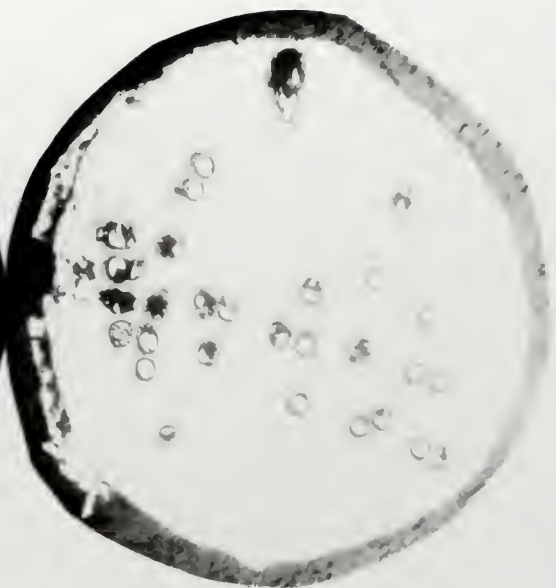


FIGURE 5. STRESS-STRAIN CURVES



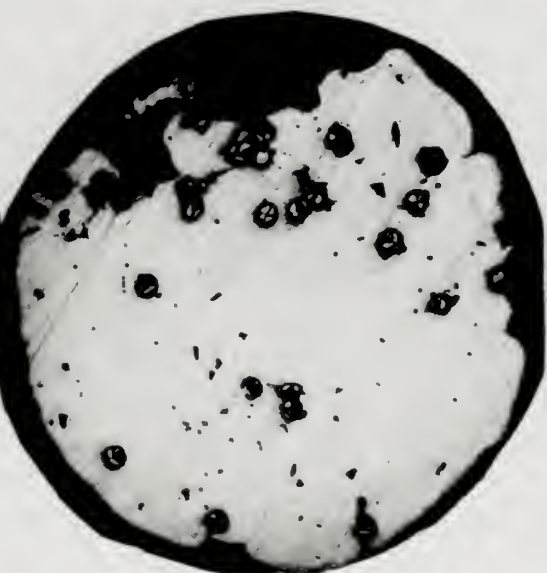
A. SPECIMEN C.



B. SPECIMEN E.



C. SPECIMEN I.

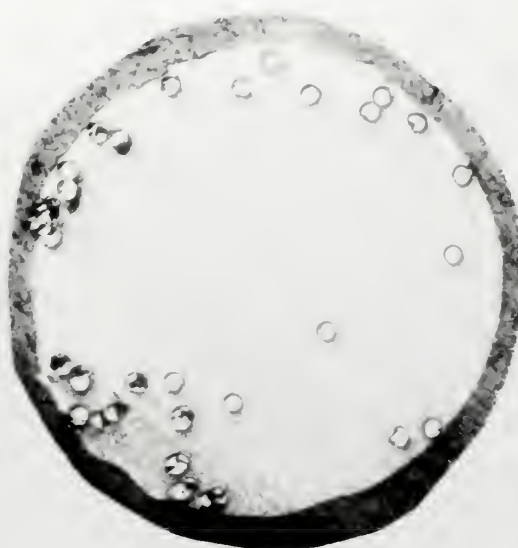


D. SPECIMEN J.

FIGURE 6. COMPLETE CROSS SECTION, 35X.



A. SPECIMEN D.



B. SPECIMEN F.

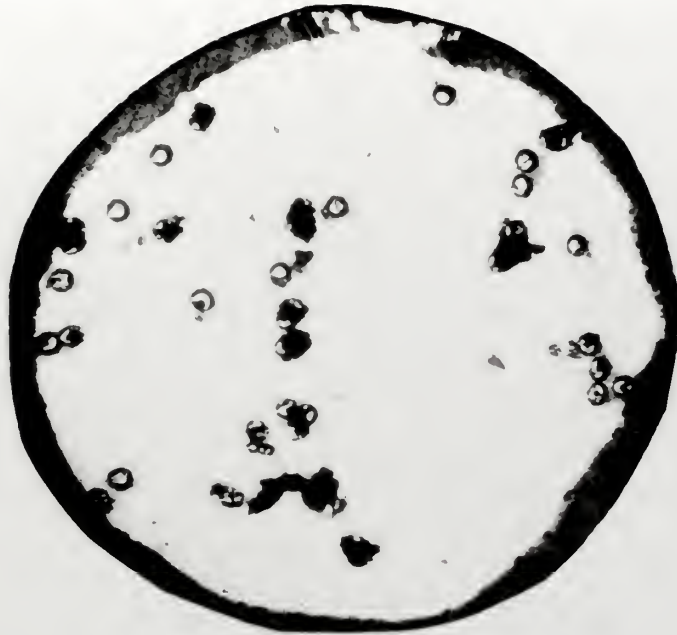


C. SPECIMEN G.



D. SPECIMEN A.

FIGURE 7. COMPLETE CROSS SECTION, 35A.



A. SPECIMEN B.



B. SPECIMEN H.

FIGURE 8. COMPLETE CROSS SECTION, 40X.

matrix such as in specimens D and F. The fiber orientation in specimen G is predominately grouped in three locations in the matrix.

The large void which can be seen in the center of specimen A was also visible in the fracture surface. The photograph of specimen J shows the poor formation of the surface of this specimen.

Figure 9 A shows the cross section of a single fiber in the matrix of specimen G. The formation of interface compound around the entire circumference of the fiber can be seen. This photograph also shows circumferential cracks in the boron filament just inside the interface compound boundary. This tendency for circumferential cracks to develop in the boron filament when interface compound formed was seen on some other photographs as well. These cracks are similar to those reported by Wolff and Hill.¹¹ The large cracks in the face of the filament are believed to have been caused during cutting and polishing operations. Figure 9 B is a single fiber in the matrix of specimen A showing the interface compound formation. Both specimens G and A were prepared with a liquid aluminum temperature of 800°C.

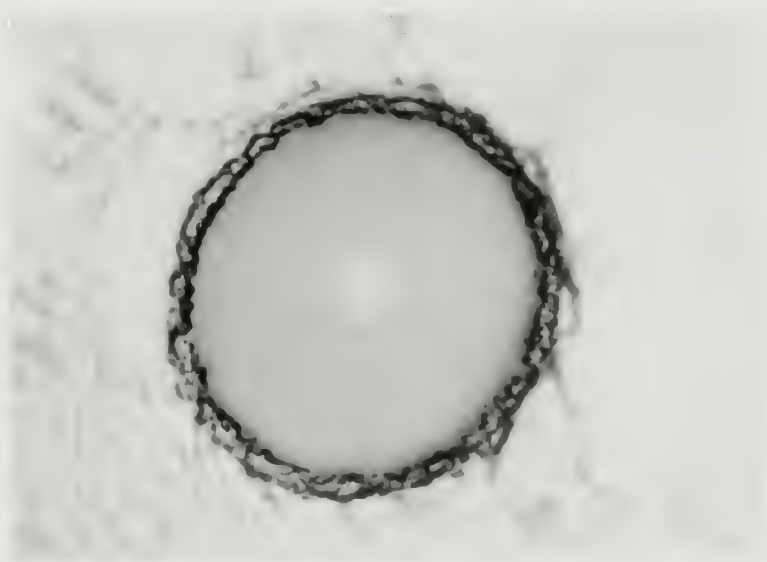
Figure 10 shows single fibers from specimens made at lower temperatures. The degree of attack on these fibers and the amount of compound formation is less than that shown in Figure 9.

Figure 11 shows groups of fibers in the matrix of specimens A and B.

A longitudinal portion of specimen G was mounted and polished and is shown in Figure 12. The specimen fracture surface is on the right. The matrix has been completely polished away from one fiber



A. SPECIMEN G.

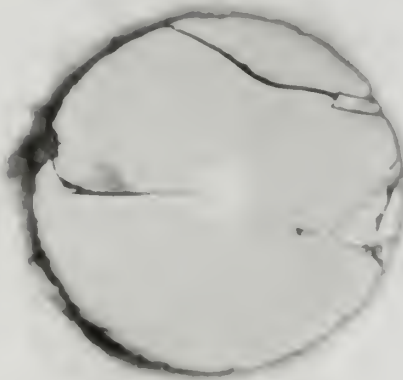


B. SPECIMEN A.

FIGURE 9. CROSS SECTIONS OF SINGLE FIBER IN MATRIX, 500X.

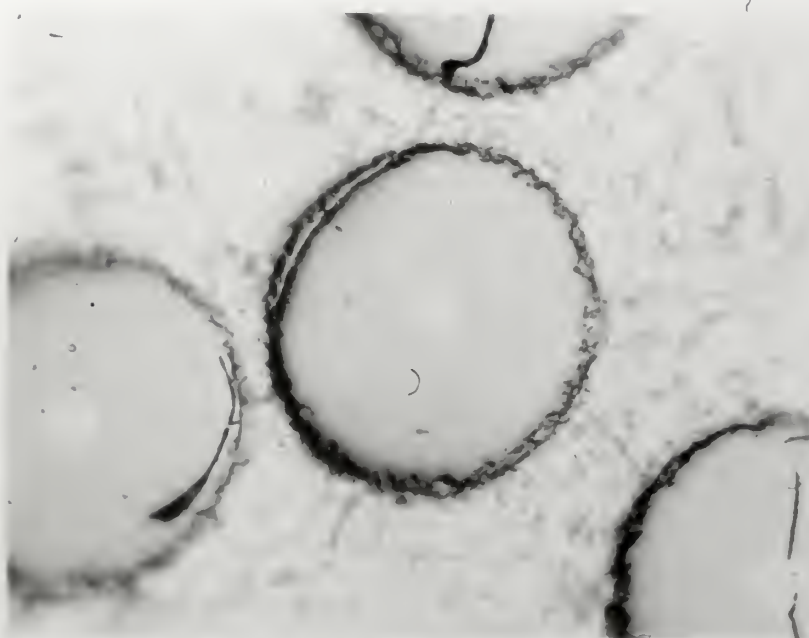


A. SPECIMEN B.

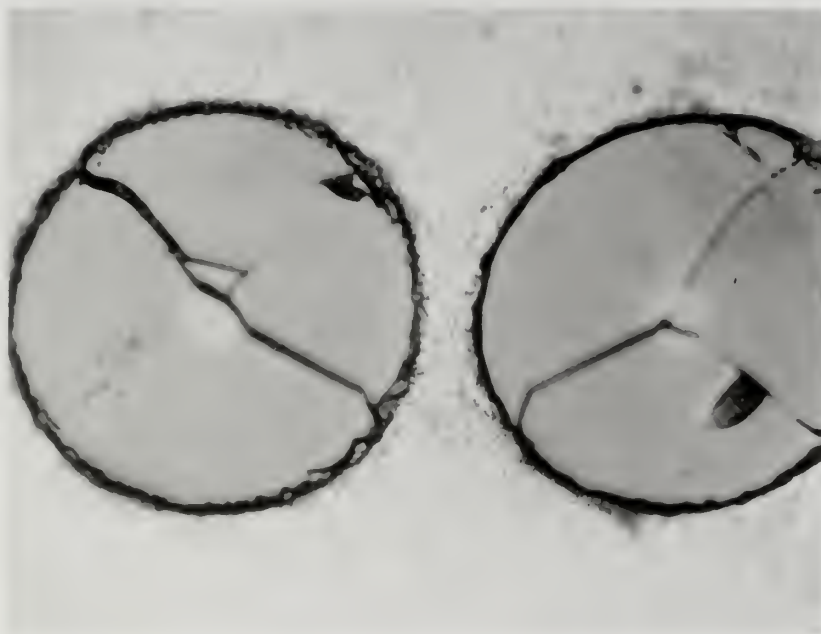


B. SPECIMEN D.

FIGURE 10. CROSS SECTIONS OF SINGLE FIBER IN MATRIX, 500X.



A. SPECIMEN A.



B. SPECIMEN B.

FIGURE 11. CROSS SECTIONS OF FIBERS IN MATRIX, 500X.

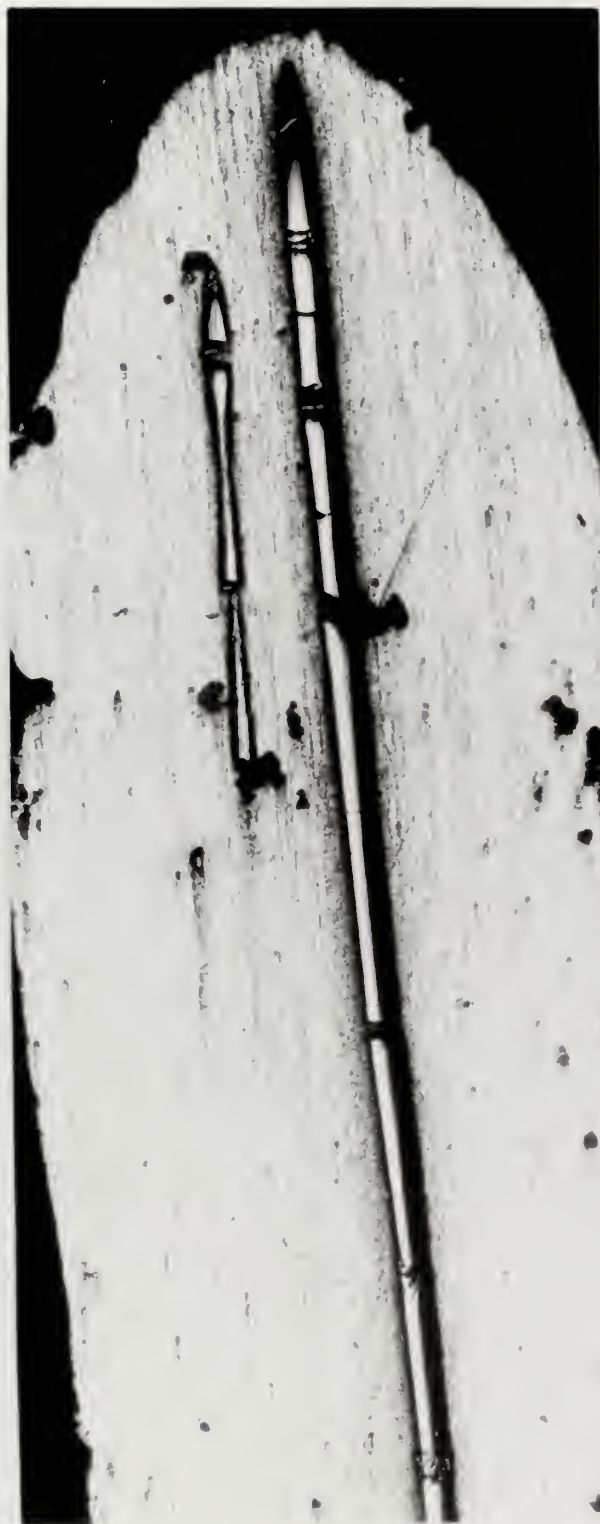


FIGURE 12. LONGITUDINAL VIEW OF FIBERS IN MATRIX, 50X

for its entire length while a second fiber is just beginning to be exposed. The numerous breaks in each fiber indicate that the fibers break at many locations in addition to the overall specimen fracture surface during tensile loading. These multibreaks in individual fibers explain the ripple trace of the Instron recorder during tensile testing. Figure 13 is the same surface after additional polishing. This figure shows that the fiber breaks in the adjacent fibers do not occur at the same longitudinal locations and that a fiber break does not cause a break in the surrounding matrix.

The view in Figure 14 is a close up of a partially polished fiber showing generally uniform formation of compound along the fiber length.



FIGURE 13. LONGITUDINAL VIEW OF FIBERS IN MATRIX, 100X.

LEFT END OF UPPER PICTURE JOINS RIGHT END OF LOWER PICTURE.

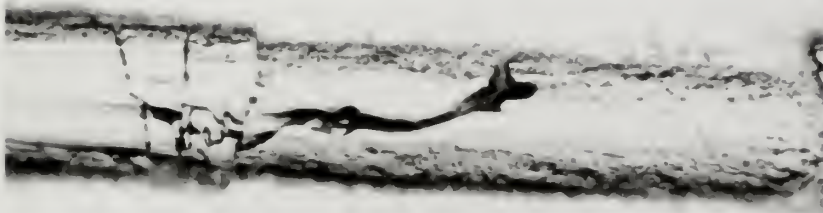


FIGURE 14. LONGITUDINAL VIEW OF FIBER IN MATRIX, 300X.
COMPOUND FORMATION CAN BE SEEN ON THE FIBER.

III. DISCUSSION OF RESULTS

A. Infiltration.

Aluminum-boron fiber composites can be prepared by vacuum infiltration if proper liquid aluminum temperatures are employed and sufficient time at infiltration temperature is allowed. This study indicated that temperatures between 740°C and 720°C for times between one and two minutes resulted in the best formed composites with the least amount of interface compound present. Higher temperatures increased compound formation and shorter immersion times resulted in poor matrix formations. All specimens prepared exhibited a reduction of fiber diameter indicating some interface compound formation at all temperatures used.

Void areas were observed in the matrix of specimens prepared at almost all temperatures employed. These voids are believed to be due to hydrogen dissolved in the liquid aluminum which reverts to gaseous form upon freezing of the aluminum. If dissolved hydrogen is indeed the reason for void formation it was most likely introduced into the liquid aluminum by moisture on and in the quartz tube containing the fibers. The methods used to preheat the tube (ie bunsen burner, 110°C oven) in this study were not good. A superior arrangement would be to have a second furnace with separate controls located above the aluminum melting furnace so that the tube could be preheated in place, while vacuum pumping, and prior to immersion. Void areas in the matrix are detrimental to composite strength since they interfere with stress transfer from fiber to fiber through the matrix.

Quartz tubes coated with carbon are considered to be less than optimum for use when infiltrating liquid aluminum because of the interaction between the aluminum and quartz. The rate of reaction increased with time and temperature. At liquid aluminum temperatures close to the melting point for short immersion times, there was very little tendency for reaction. Vycor tubes exhibited this same phenomenon. Furthermore, traces of a yellow cast were noted on the frozen remains of the crucible after an infiltration was completed. These traces were believed to be silicon removed from the glass during aluminum attack.

The graphite crucible used to hold the liquid aluminum showed no tendency toward reaction. On this basis, graphite would be an excellent material for infiltration tubes. Unfortunately, attempts to make or locate a source of supply for graphite tubes of the required dimensions met with no success during the time allotted for this study.

Since solid aluminum is relatively non-reactive with boron, in a reasonable laboratory time, it is important that apparatus be available which will not react with liquid aluminum if aluminum boron composite interface compound studies are to be conducted.

B. Composite Strength.

Of the ten composites tested, all showed a marked departure from a nominal strength predicted by the law-of-mixtures. The amount of this strength reduction which can be attributed to the interface compound and that which can be attributed to other factors cannot be determined from this study. The strength reduction role played

by such factors as fiber orientation, fiber spacing, notches due to compound formation, matrix voids, and fiber alignment could not be quantitatively determined. Another factor to be considered is the actual strength of fibers in a given composite, especially a low density composite. Estes⁸ tensile tested twelve individual boron filaments cut from a United Aircraft roll finding that four failed below 70,000 psi and the remaining eight averaged at 327,500 psi. In the work done by Wolff and Hill¹¹ they indicated that as received boron filaments used in some of their samples had a strength range of from 72,000 psi to 423,000 psi.

A review of Tables I and II indicates that specimen strength is generally reduced by higher infiltration temperatures and times and that interface compound increases with higher temperatures and times. Figure 15 is an attempt to find a relative effect on maximum tensile strength of interface compound within the group of ten specimens tested. The ordinate of this plot is specimen maximum tensile strength normalized to that strength predicted by the law-of-mixtures for an "ideal" composite of 0.087 inches in diameter containing thirty 0.00395 boron filaments of 300,000 psi ultimate tensile strength. This value is denoted by G_c/G_r . The abscissa is the measured fiber diameter squared divided by the specimen measured diameter squared. This is denoted by $(d'/D)^2$. The latter value is representative of the volume fraction of available fiber after interface compound formation. The line shown on the plot as "Ideal Nominal Strength" is for the "ideal composite containing fibers of reducing diameter. A line running parallel to the "Ideal Nominal Strength" line and through the plot for specimen E follows the tensile test

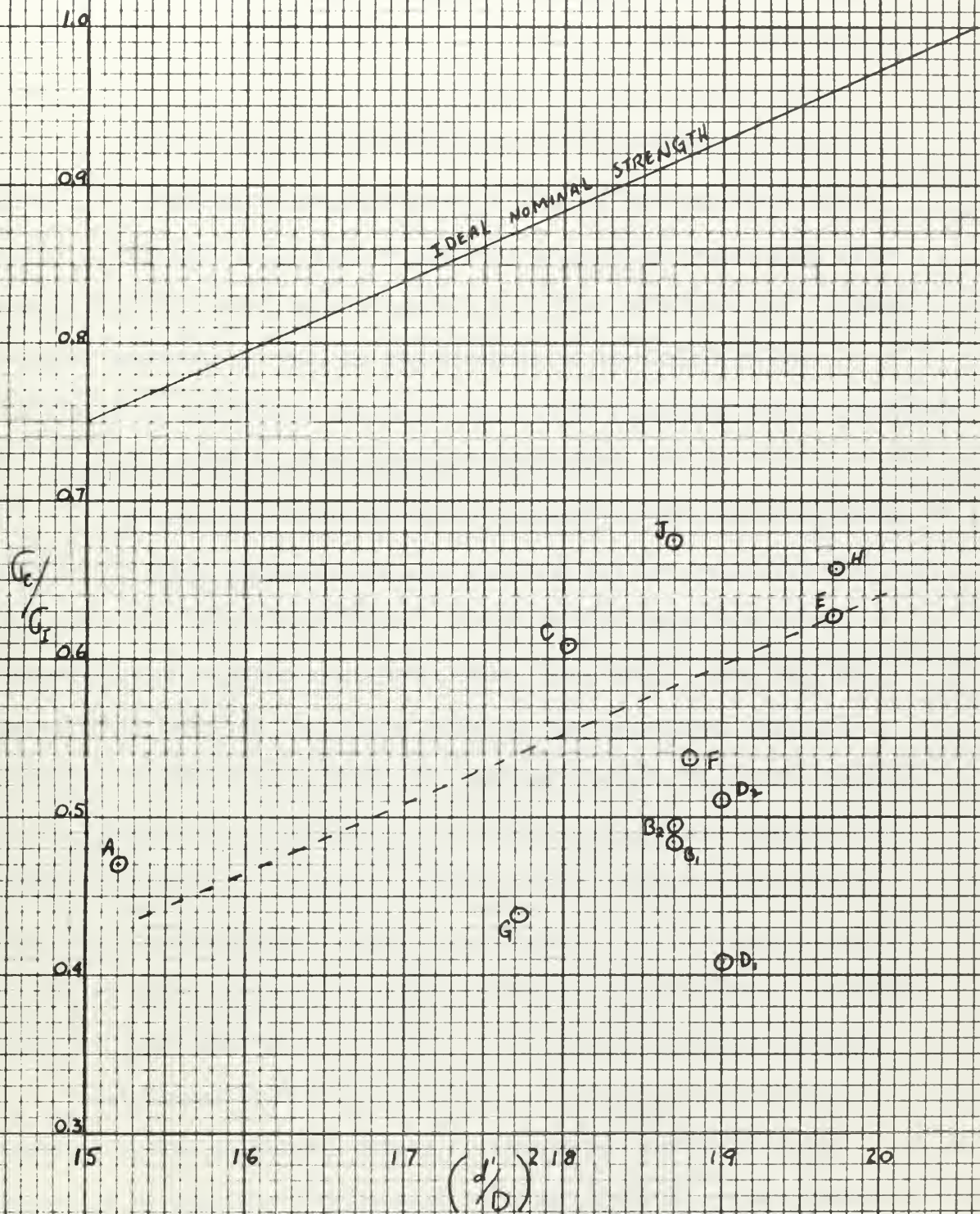


FIGURE 15. Curve of Normalized Strength to Diameter Ratio Squared.

data if the plot of specimen I is discounted. This indicates that within the test group a modified law-of-mixtures rule, which neglects the volume fraction made up by interface compound, can be relied upon to estimate the relative strengths of the specimens within the group. Evaluation of the total real effect of the interface compound on strength will require specimen preparation techniques which yield specimens more closely approaching ideal configuration. Higher density specimens as well as improvements in the infiltration techniques as previously discussed may aid in producing better specimens.

C. Multiple Fiber Breaks and Composite Failure.

For the volume fraction used, approximately 0.06 in all composite specimens, fiber strengthening was realized and the composites did not fail when a single fiber failed. Matrix shear continued to load broken fibers to produce multiple breaks in a single fiber without necessarily lowering the stress level on the composite. Multiple breaking of fibers is expected to continue until broken fiber lengths are diminished to a length equal to twice the critical length. The critical length of a fiber is defined as the minimum length needed to achieve maximum fiber stress at one point.¹ The multiple breaking of fibers in the composites tested indicates a good degree of bonding between fibers and matrix even with the interface compound present.

Final composite failures occurred in a single cross section by fracture of the matrix and fibers at that cross section with the exception of specimen I where poor fiber-matrix bonding was present. The ideal failure cross section is related to critical length in that it will occur where fiber breaks accumulate within a distance of one

critical length and the remaining sufficiently long fibers can no longer support the load.

IV. CONCLUSIONS

A. Aluminum-boron fiber composited prepared by infiltration:

1. The best formed composites with the least amount of interface compound were prepared at liquid aluminum temperatures in the range of 720°C to 740°C for infiltration times between one and two minutes.
2. Interface compound formed reducing fiber diameters when boron was in contact with liquid aluminum for any period of time.
3. Interface compound formation and fiber degradation increased with infiltration time and temperature.
4. Aluminum in the solid state is relatively non-reactive with boron.

B. Composite strength with interface compound present:

1. The composites tested followed a modified law-of-mixtures rule which neglects the volume fraction of interface compound and accounts for only actual fiber and matrix volume fractions, however, the test strengths were only $2/3$ of that predicted. This variation in strength is attributed to the following factors:
 - a. Fiber orientation and fiber spacing.
 - b. Voids and imperfections in the matrix.
 - c. Notches in fibers due to compound formation.
 - d. Variation in individual fiber strengths.
2. Interface compound formation did not prevent fiber-matrix bonding nor multiple single fiber breaking as a result of matrix shear loading.

V. RECOMMENDATIONS

1. Vacuum infiltration techniques for aluminum-boron composites should be investigated from a metallographic standpoint in an effort to approach ideal composite configuration.
2. Interface compound effects using aluminum-boron composites in the 0.30 fiber volume fraction range should be studied. Machine cutting of the fibers to the desired length would greatly aid in accomplishing this.

BIBLIOGRAPHY

1. Broutman, L. J. and Krock, R. H., Principles of Composites and Composite Reinforcement, Modern Composite Materials, Addison-Wesley, 1967, pp. 3-26.
2. Weeton, J. W. and Signorelli, R. A., Fiber-Metal Composite Materials, NASA TN D-3530, August 1966.
3. M^cDanels, D. L.; Jech, R. W.; and Weeton, J. W., Metals Reinforced With Fibers, Metal Progress, Vol. 78, No. 6, December 1960, pp. 118-121.
4. Petrasek, D. W., Elevated-Temperature Tensile Properties of Alloyed Tungsten Fiber Composites, NASA TN D-3073, October 1965.
5. Heitman, P. W., Fracture of a Molybdenum-Aluminum Fiber Composite, PhD Thesis, Department of Metallurgy and Materials Science, 1968.
6. Wawner, F. E., Jr., Boron Filaments, Modern Composite Materials, Addison-Wesley, 1967, pp. 244-269.
7. Metals Handbook, Vol. 1, 8th Ed., American Society for Metals.
8. Estes, C. D., Formation of Boron Fiber-Aluminum Composites by Drawing Processes, Engineer Thesis, Department of Naval Architecture and Marine Engineering, 1966.
9. Camahort, J. L., Protective Coating by Surface Nitridation of Boron Filaments, Journal of Composite Materials, Vol. 2, No. 1, January 1968, pp. 104-112.
10. Alexander, J. A., The Elevated Temperature Reactivity of Boron-Metal Matrix Composite Materials, General Technologies Corp., Reston, Virginia. Summary Technical Report AFML-TR-67-101, Contract AF 33(615)-3155, July 1967, AD 820900.
11. Wolff, E. O. and Hill, R. J., Research on Boron Filament/Metal Matrix Composite Materials, Report AFML-TR-67-140, Space Systems Division, AVCO Corp. Lowell, Mass. Contract AF 33(615)-3164 June 1967, AD 816439.
12. Hanby, K. R., Fiber-Reinforced Metals, DMIC Review of Recent Developments, October 9, 1968.
13. Riley, A. A., An Inch-Wide Rope Capable of Lifting 4-Engine Jet, Boston Sunday Globe, January 26, 1969, pp. A-8.
14. Opportunities for Naval Research, ONR-17, (Rev. 7-68), Department of the Navy.

15. Trozera, T. A.; Sherby, O. D.; and Dorn, J. E., Effect of Strain Rate and Temperature on the Plastic Deformation of High Purity Aluminum, American Society for Metals Transactions, Vol. 49, 1957, pp. 173.

thesW5508

The effect of the interface compound on



3 2768 001 95054 6

DUDLEY KNOX LIBRARY